International Journal of Mechanical and Production Engineering Research and Development (IJMPERD)
ISSN (P): 2249-6890; ISSN (E): 2249-8001
Vol. 9, Issue 4, Aug 2019, 425-436


# EIGN-LIMITS UNCERTAINTY METHODOLOGY FOR TEMPERATURE CALIBRATION BY FIXED POINTS AND COMPARISON

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***ABSTRACT***

***Calibration measurement uncertainty of Platinum Resistance Thermometer (PRT) was estimated in International Temperature Scale 1990 (ITS-90) at defined fixed points [1]but it was not gives a criteria to find the uncertainty at un-calibrated point within sub ranges between fixed points experimentally, the uncertainty was estimated mathematically. This paper explains the estimation of measuring uncertainty for PRT based on mathematical module able to detect the uncertainty at specified un-calibrated temperature (Intra limits propagation) and the expectation for the Eign-limits using modified supplementary ITS-90equation, this equation was used by modification the factor of sensitivity*** $f_i^2$***according to each uncertainty source. It was covered the range from 0°C to 420°C. (Water Triple Point (WTP) 0.01 °C, Gallium Melting Point (GMP) 29.76 °C, Tin Freezing Point (SnFP) 231.9 °C and Zinc Freezing Point (ZnFP) 419.5 °C). Same range was covered in the calibration by comparison method. From this study, a validation for the equation has carried out, the uncertainties in the sub ranges were found to be less than 0.2 mK difference between the real measurements and statistically expectation.***

***KEYWORDS: Uncertainty, Comparison, Fixed Point & ITS-90***

**Received**: Apr 12, 2019; **Accepted**: May 02, 2019; **Published**: Jun 29, 2019; **Paper Id.**: IJMPERDAUG201942

**Original Article**

## 1. INTRODUCTION

One of the fundamental missions of the thermal metrology laboratory in NIS-Egypt is to realize ITS-90 [1]. The measurement uncertainty in the calibration of a PRT depends on the calibration method used, the uncertainty contribution of the standards, the characteristics of the measuring equipment used and the characteristics of the device under calibration. No general instructions for the estimation measurement uncertainty in the subranges. PRT was calibrated using defined fixed points according to ITS-90 at (WTP), (GMP), (SnFP) and (ZnFP). PRT was calibrated also by comparison in the same range from 0.01 °C up to 419.5 °Cusing temperature-stabilized baths model, fluke standard thermometer, resistance measuring bridge model ASL F-18[2,3] which have been traceably calibrated. In this study, the estimation of measurement uncertainty at calibrated points and the estimation of sub ranges measurement uncertainty by using modified ITS supplementary equation was done. Also, comparing difference between the real and statistical evaluation.

## 2. EXPERIMENTAL WORK

Two calibration methods were used in this work.

### 2.1 First Method (Calibration at Fixed Points)

It was carried out according to the requirements of ITS-90 (1). Four fixed point cells were used in this work to cover the range from 0.01 °C up to 419.5 °C. Figure 1 shows schematic diagram of the fixed point

measuring system assembly.(WTP), (GMP), (SnFP) and (ZnFP) were measured according to references (1&7).

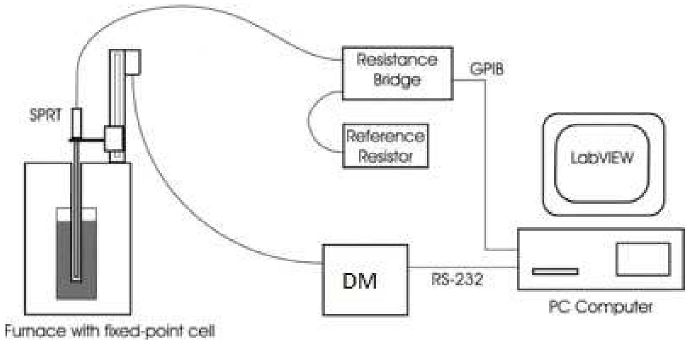


**Figure 1: Fixed Point Measuring System Assembly**

### 2.2 Second Method (Comparison Calibration)

It is performed by measurement of the resistance of PRT under calibration while it is exposed to a temperature. Figure 2 shows schematic diagram of the comparison calibration system. A calibration bath cannot be considered as completely stable in time and homogeneous all over its volume, especially when temperature calibrations by comparison are performed at the best level of uncertainty. This represents a major contribution to the total uncertainty of a calibration procedure. In order to decrease this uncertainty contribution equalizing control [4] can be used in calibration baths. A gradient is observed as a change of a temperature reading of a PRT according to a change of its position inside a calibration bath. Basic gradients that can be observed are vertical and horizontal gradient. Uncertainty contribution of an axial gradient is determined as maximum temperature difference between two different positions in axial direction. The radial gradient is a maximum temperature difference between two different positions in a radial direction. A short-term stability of a medium temperature depends on type of regulation and flow of medium inside the bath. Since the calibration measurements were taken within short time interval, the short-time stability is relevant (30min). For the time stability of a bath, temperature deviations of a reference thermometer are observed. Stable temperature mediums is working in the range from 0.01 °C to 419.5 °C, data acquisition is performed via multimeter and GPIB interface or RS232 interface. Environmental conditions (temperature and relative humidity)was monitored during calibration.

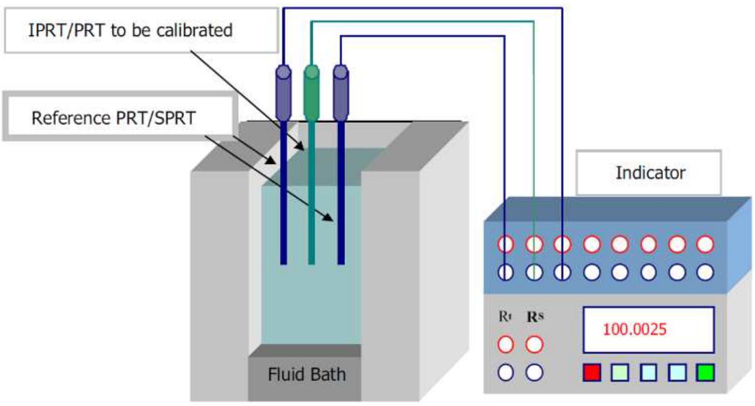


**Figure 2: Comparison Calibration System Assembly**

## 3. UNCERTAINTY METHODOLOGY

### 3.1 Uncertainty of Fixed Point Calibration

All of the ITS-90 interpolating equations can be expressed as a sum of interpolating functions multiplied by the corresponding reference resistance ratios:

$$W_r(W) = \sum_i^n w_{r.i} f_i(W) \tag{1}$$

Where the numerical index *i* refers to the sequence of fixed points used to calibrate the SPRT (e. g. WTP, Sn, Zn, etc). The empirical data itself fitted according to the equations of ITS-90 equation. The fitted data was polynomial from 15th degree, the uncertainty estimated from regression of the error as shown in figure 3.

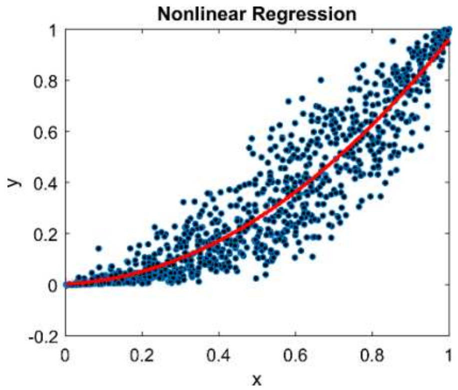


**Figure 3: Non-Linear Regression for the Estimator**

Differentiation of with respect to all 2*n* + 1 parameters leads to the most general form of the propagation of error equation, as shown in equation 2.

$$dw_r = \sum_{i=1}^n f_i(W) dW_{r.i} - \sum_{i=1}^n \left(f_i(W) \frac{\partial W_r}{\partial W}\Big| W_i\right) dw_i + \frac{\partial W_r}{\partial W} dw \tag{2}$$

The uncertainties associated with each resistance measurement are not correlated so the contributing uncertainties due to the WTP cells and resistance measurements are independent on by another meaning uncorrelated with week covariance. The corresponding expression for the uncertainty in the intera *W*r value is given as the following equation and propagation as mentioned in ITS-90 text, as shown in equation 3 and figure 4 and 5.

$$U_{E-L}^2(W_r) = \frac{1}{R_{WTP}^2}\left((u^2(R) + w^2 u^2(R_{WTP})\right) + \sum_{i=2}^n f_i^2(w)\left(u^2(R_i) + W_i^2 u^2(R_{WTP.i})\right) \tag{3}$$

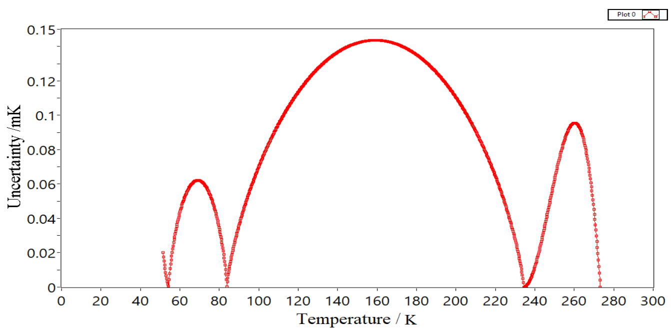


**Figure 4: Interpolating Expectation values for Uncertainty Propagation**

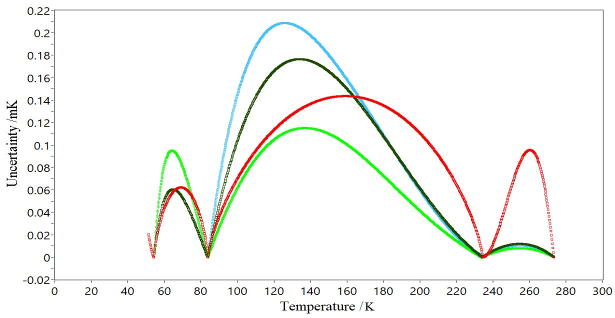


**Figure 5: Interpolating Expectation Values for Uncertainty Propagation within SPRT Subranges**

There are several sources of uncertainty that related to the SPRT and fixed point. The estimation here based on the most difficult sources to be estimated that related to the realization of fixed point. Those sources have been addressed and estimated at each point. The following contributing sources are:

### 3.1.1 Uncertainty from Standard Resistor and Oil Bath

The two main contributions to the uncertainty in the value of the standard resistance can be evaluated using a simple model for the temperature dependence of the resistance:

$$R_s(t_{bath}) = R_s(t_{cal})[1 + \beta(t_{bath} - t_{cal})] \tag{4}$$

Where

$t_{bath}$ is the temperature of the oil bath used to maintain the resistor during use and its temperature set-point was adjusted at 23.00 $^{o}$C according to manufacture calibration certificate and instructions for the standard resistor., $t_{cal}$ is the temperature of the bath used to maintain the resistor when it was calibrated, and β is the temperature coefficient of the resistor. The temperature coefficient is normally expressed as the fractional change per degree; i. e,

$$\beta = \frac{1}{R_s}\frac{dR_s}{dt} \tag{5}$$

With typical values for good-quality resistors within the range ±5 × $10^{-6}$ /ºC.

The cumulative uncertainty in the resistance value due to these terms, when β ($t_{bath}$-$t_{cal}$) << 1, is:

$$u^2(R_s) = u^2(R_{s,cal}) + R_s^2\left[\beta^2 u^2(t_{bath}) + (t_{bath} - t_{cal})^2 u^2(\beta)\right] \tag{6}$$

where u ($R_{S,\ cal}$) is the calibration uncertainty of the resistor as supplied by the electrical calibration laboratory, u($t_{bath}$) is the uncertainty arising from fluctuations in the resistor-bath temperature, and u(β)is the uncertainty in the temperature coefficient of the resistor. Where the $t_{bath}$ and $t_{cal}$ are nominally equal, the uncertainty in the temperature coefficient can usually be ignored.

### 3.1.2 Uncertainty from Hydrostatic Pressure

The practical realization of the fixed points, with a thermometer well surrounded by at least one solid-liquid interface, results in a vertical pressure gradient along the length of the thermometer well with the pressure determined by the depth of liquid above the sensing element of the thermometer. The measured temperature must be corrected for the

hydrostatic pressure difference between the surface of the liquid, where the fixed-point temperature is defined, and the thermal center of the sensing element of the SPRT.

$$\Delta T_{hyd} = -\frac{dT}{dh}(h_{liq} - h_{SPRT}) \tag{7}$$

Where

$h_{liq}$: is the vertical elevation of the surface of the molten material in the cell when the measurement is taken.

$h_{SPRT}$: is the vertical elevation of the thermal center of the sensing element.

dT/dh: is the hydrostatic-pressure coefficient defined by ITS-90, the hydrostatic elevation and pressure are related by

$$\frac{dT}{dh} = \rho g \frac{dT}{dP} \tag{8}$$

Where ρ is the density of the liquid and g is the acceleration due to gravity. The uncertainty in the hydrostatic correction is given by

$$u^2(\Delta T_{hyd}) = \left(\frac{dT}{dh}\right)^2 \left[u^2(h_{liq}) + u^2(h_{SPRT})\right] + u^2\left(\frac{dT}{dh}\right)(h_{liq} - h_{SPRT})^2 \tag{9}$$

Where u ($_{hliq}$) and u ($h_{SPRT}$) are the uncertainties in the elevations, and u (dT/dh) is the uncertainty in the hydrostatic pressure coefficient.

### 3.1.3 Uncertainty from Self-Heating Correction

When there is a temperature distribution inside the block (metal ingot), the propagation of the heat wave, generated by the excitation current of the SPRT, becomes asymmetric, when more thermal energy propagates along the flux and less energy propagates against the flux. The measurement of resistance necessarily involves passing a current through the resistor, with a consequent dissipation of heat, which results in a heating of the sensor wire above the medium temperature. According to Fourier law, the thermal energy exchanged between the sensing element, at a temperature T, and the medium temperature $T_o$ is given by:

$$\varphi_i = \frac{T - T_o}{R_{th}} \tag{10}$$

Where:

- $\varphi_i$ is the thermal energy exchanged between sensing element.
- $T_o$ is the measuring temperature of cell.
- T is the measuring temperature of the SPRT.
- $R_{th}$ is the thermal resistance between the sensing element and the medium temperature under study.

Practically, $R_{th}$ contains several thermal resistances such as resistances of, thermometer filling gas, thermometer sheath, the medium between the thermometer sheath and cell well itself. Thermometer self-heating corresponds to the temperature difference ($T-T_o$), where;

$$(T - T_o) = R_{th}.\varphi_i = R_{th}.R.I^2 \quad (11)$$

The difference depends on the electric resistance, R, of the thermometer (at the considered temperature), the measuring current, I, and the thermal resistance, $R_{th}$. thus the self-heating not only depends on the technology used to fabricate the thermometer, but also on the medium characteristics (geometrical and physical), where the thermometer is immersed. This correction is always associated with an uncertainty, so the used currents are chosen to be 1 mA and √2 mA which are a good compromise between the self-heating value and the bridge sensitivity. The value of this uncertainty is calculated as a contribution of the dispersion of the ratio between the two measuring currents, and of the bridge resolution.

### 3.1.4 Long Term Drift

Oxidation, impurities, and crystal defects all give rise to an increase in the triple-point resistance of an SPRT, and all may occur during the use of the SPRT. Because the three effects have a similar effect on the triple-point resistance, but have different propagation laws, increases in the triple-point resistance necessarily give rise to uncertainty (ambiguity). Long-term drift in SPRT is evident from changes in the $R_{WTP}$ values that cannot be removed by annealing. The shifts tend to be a mix of two types of change. The drifts may be due to insufficient annealing during manufacture. Secondly, permanent dimensional changes, caused by plastic deformation, volatilization of platinum lead to a change in the triple-point resistance.

Ideally where thermometers exhibit significant drift, they should be recalibrated. Also, drift caused by impurities tends to accelerate with time, so excessive drift usually suggests replacement rather than recalibration. By another meaning, the SPRT calibrated at WTP then annealed then calibrated again at WTP periodically once per year and taking into account the values of $R_{wtp}$ to find the differences and drift. Uncertainty value (assuming a rectangular distribution) related to long term drift is:

$$u^2\left(\Delta W_{drift}\right) = \frac{u^2\left(\Delta R_{WTP,drift}\right)}{\left(R_{WTP}\right)^2}\left(1 - W\right)^2 \quad (12)$$

An example for the uncertainty budget for the SPRT that calibrated at any point has shown in table 1

**Table 1: Uncertainty Sources related to SPRT**

| Source | Value / K |
|---|---|
| Bridge | 5.41E-06 |
| St. Resistor & Oil Bath | 0.00008 |
| Impurities and oxidation | 7.00E-06 |
| Hydrostatic | 3.00E-05 |
| Self-heating | 2.00E-06 |
| long term drift | 0.00003 |
| Heat flux | 0.00023 |
| Calibration | 1.00E-04 |

$$U = \sqrt{\begin{matrix}(5.4x10^{-6})^2 + (8\,x10^{-5})^2 + (7x10^{-6})^2 + (3x10^{-6})^2 \\ + (2x10^{-6})^2 \; + (3x10^{-5})^2 + (2.3x10^{-5}\;)^2 + (1x10^{-4}\;)^2\end{matrix}} = 0.27 \text{ mK}$$

### 3.2 Uncertainty of Comparison Calibration

The temperature, at which the calibration item is calibrated, is determined by measurement with the standard thermometer and by additional corrections:

$$Tc = Tr + \delta T_{acqTr} + \delta T_{acqTc} + \delta T_{bath} \tag{13}$$

Where:

Tc is temperature of the thermometer to be calibrated, Tr is the temperature of the reference thermometer, $\delta T_{acq\ Tr}$ is linked with the acquisition of reference thermometer (bridge, multimeter…), δTacq Tc is linked with the acquisition of calibrated thermometer (bridge, multimeter…) and δT bath is linked with the bath (homogeneity, time stability). The repeatability of the measurements (Figure 6, 7 and 9) is consider the major part in our study. The repeatability could be analysis statistically either by the expectation value of median or by portability distribution function with respect to sigma (standard deviation). The uncertainty propagation shown in figure 7.

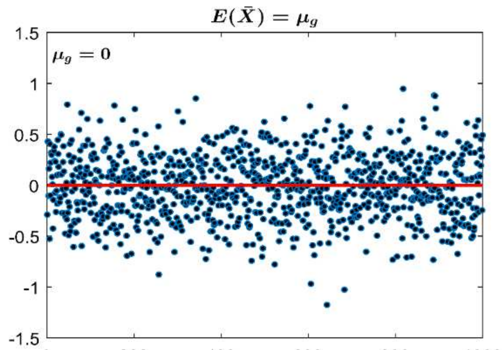


**Figure 6: Uncertainty Estimator for Expectation value for Comparison Calibration due to Repeatability**

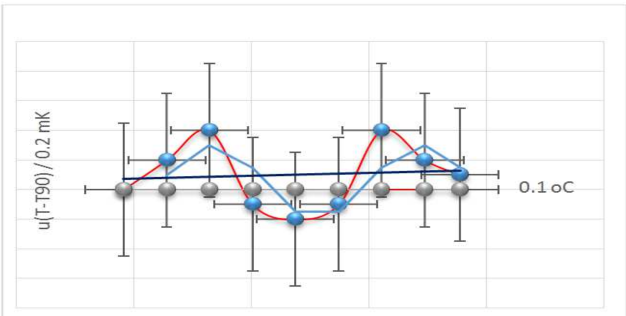


**Figure 7: Uncertainty Propagation for Comparison Calibration due to Hysteresis**

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Uncertainty propagation equation for uncelebrated temperature point used in the calibration by comparison.

$$T = f(R).u_c^2(T) = \sum_{i=1}^{N}\left(\frac{\partial f}{\partial x_i}\right)^2 u^2(x_i) + 2\sum_{i=1}^{N-1}\sum_{j=i+1}^{N}\frac{\partial f}{\partial x_i}\,\frac{\partial f}{\partial x_j}\,u(x_i)u(x_j)\,\rho_{ij} \tag{14}$$

### 3.2.1 Uncertainties Components Linked to Reference Standard - $T_r$

$u_{Trc}$ is combined uncertainty and $U_{tr}$ is expanded uncertainty. It was found in the calibration certificate of the reference standard. It presents as:

$$u_{Trc} = \frac{U_{Tr}}{2} \tag{15}$$

### 3.2.2 Drift

The temperature drift and the systematic biased is considered to be rectangular distribution that has limited value governed the estimated behaviour (figure 8).

$$u_{Trd} = \frac{DriftValue}{\sqrt{3}} \tag{16}$$

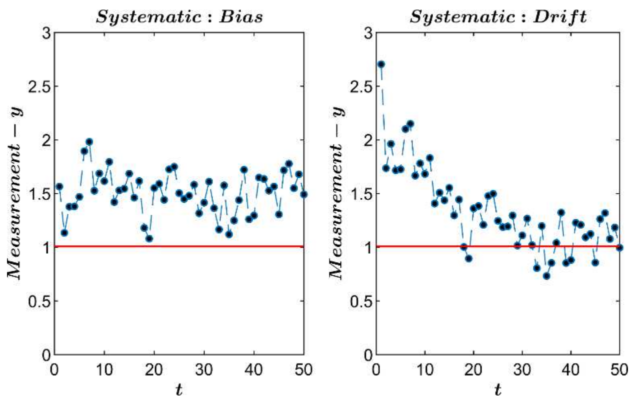


**Figure 8: Uncertainty Estimated form Drift and Biased**

### 3.2.3 Repeatability

$u_{Tra}$ is the repeatability of measurements during a calibration. We assume at least 50 measurements of reference standard was made at each calibration point. Standard deviation of mean was calculated. The median is the major parameter to satisfying the best repeatable value within sigma (δ) equivalent to 95% confidence level.

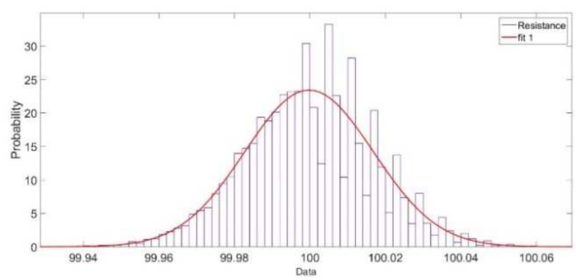


**Figure 9: Uncertainty Estimated form PDF Repeatability**

### 3.2.4 Self Heating Effect

$u_{Trsh}$ is uncertainty component due for self-heating of the reference standard. The measurement of resistance involves passing a current through the resistor and, therefore, heating of the resistor. For the highest accuracy measurements, corrections are applied by measuring at two currents, I1 and I2 (1 mA and √2 mA), and extrapolating to

zero current [4, 5, 6]. For the determination of the electrical resistance, an electrical measurement must be carried out for which a measurement current must be fed through the sensor (figure 5).

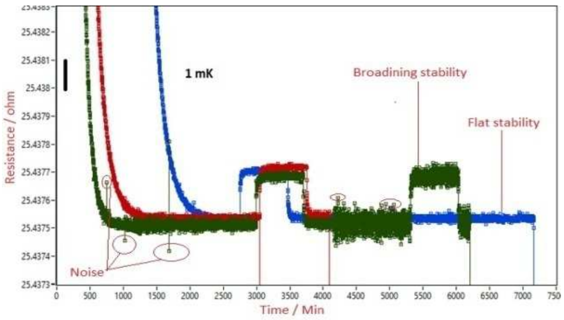


**Figure 10: Self-Heating Effect**

The measurement current leads to the sensor being heated (self-heating) and thus to the measurement result being falsified. This effect is dependent not only on the magnitude of the measurement current but also on the measurement conditions themselves. In the calibration, the self-heating mechanism is to be investigated or a measurement current is to be chosen at which this effect is negligible, resistance at zero current.

### 3.2.5 Uncertainties Linked to Unit under Calibration -Tc

$u_{Tca}$ is the repeatability of measurements during a calibration. The results were taken 50 measurements of instrument under calibration was made at each calibration point. $u_{Tch}$ is the uncertainty component due to hysteresis. In general, hysteresis is a phenomenon that results in a difference in an items behaviour when approached from a different path. In PRTs, thermal hysteresis results in a difference in resistance at a given temperature based on the thermal history to which the PRT was exposed. More specifically, the resistance of the PRT will be different when the temperature is approached from an increasing direction vs a decreasing direction, and the magnitude of the difference will depend on the magnitude of the temperature excursion and the design of the PRT. Let that $u_{Tccon}$ is the uncertainty contribution due to a possible heat conduction by the instrument under calibration. Tests should be made at different immersion depths. Pulling the instrument 20 mm out of the bath led to a temperature change of 2 mK (which due to the temperature variations of the bath could be estimated only inaccurately).

### 3.2.6 Data Acquisition

Two multimeters used for data acquisition for reference thermometer and instrument under calibration, so the uncertainty contribution must be accounted for reference thermometer and device under calibration, let that $u_{Rohm}$ is the uncertainty contribution due to measurement uncertainty in the calibration of the thermometry bridge.

According to the calibration certificate, the measurement uncertainty of the multimmeter is 0.020 Ω (k = 2) and the standard uncertainty thus is 10 mΩ. $u_{Rts}$ is the uncertainty contribution due to time stability of multimeter (user manual). $u_{Rt}$is the uncertainty contribution due to ambient temperature influence (user manual). Outside certain ambient temperature interval, the component is significant and can be accessed using user manual. Within prescribed temperature interval the component is negligible. $u_{Rres}$ is uncertainty component due to multimeter resolution – least significant bit LSB. The limited resolution of the bridge of 0.001 Ω allows a reading within ±0.0005 Ω. From this a standard uncertainty of 0.5 mΩ / √3 = 0.29 mΩ, figure (7). Uncertainty linked to the connection, the reference probe is four wire connected to multimeter and the uncertainty associated with this connection type is taken to be negligible.

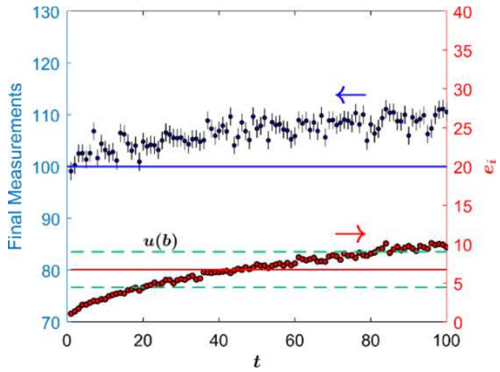


**Figure 11: Equipment Uncertainty Effect**

### 3.2.7 Uncertainties Components Linked to Temperature Fixed Point Furnace– $T_{oven}$

The spatial and temporal temperature distribution in the working space of temperature stabilized bath must be quantitatively determined and taken into account for uncertainty budget evaluation. Method for the determination of the temporal and spatial distribution involves calibrated thermometers of identical type, positioned on the boundaries of the working space (horizontal, vertical) of the temperature bath. After thermal stabilization, the temperatures measured with the thermometers are continuously recorded (typically over a period longer than 30 min). The maximum temperature difference between the thermometers is used for as uncertainty component in the uncertainty budget (rectangular distribution). Temperature gradients in temperature-stabilized baths or furnaces can be reduced by providing a metallic stabilizing block with holes to accommodate the standards and calibration items. The thermometer calibration may begin after both the temperature is stabilized bath and the thermometer itself have reached thermal equilibrium. Uncertainty contribution of an axial gradient is determined as maximum temperature difference between two different positions in axial direction. The radial gradient is a maximum temperature difference between two different positions in a radial direction. $U_{Oh}$is Spatial homogeneity is a gradient is observed as a change of a temperature reading of a thermometer according to a change of its position inside a calibration bath. Basic gradients that can be observed are vertical and horizontal gradient but sometimes more appropriate to define axial and a radial gradient. Uncertainty contribution of an axial gradient is determined as maximum temperature difference between two different positions in axial direction. The radial gradient is a maximum temperature difference between two different positions in a radial direction. $U_{Os}$ is temporal stability, important characteristic of a bath is also short-term stability of a medium temperature. It strongly depends on type of regulation and flow of medium inside the bath. Since the calibration measurements are taken within short time interval, the short-time stability is relevant. For the time stability of a bath, temperature deviations of a reference thermometer were recorded.

## 4. EXPERIMENTAL VALIDATION

Several runs have been carried out to validate the temperature uncertainties on un-calibrated point to verify the equation. The selected point was at temperature 200 $^0$C, in the subrange from the triple point of water up to freezing point of tin (0.01$^o$C to 231.927 $^0$C) the uncertainty associated to this range could be estimated from the following propagated equation as

$$U_t= (1.4974 \times 10^{-5}\, t)\,(231.928 - t) \tag{17}$$

The standard uncertainty is found to be 4.7 mk which gives good agreement with that calculated from real realization. Figure 12 illustrate the different between two uncertainties from mathematical expectation (1), it is found to be

4.7 mk and that comes from real realization (2) which is found to be 4.5 mk as shown in figure 12 and table 2.

Note: The calculation must be more to cover the range

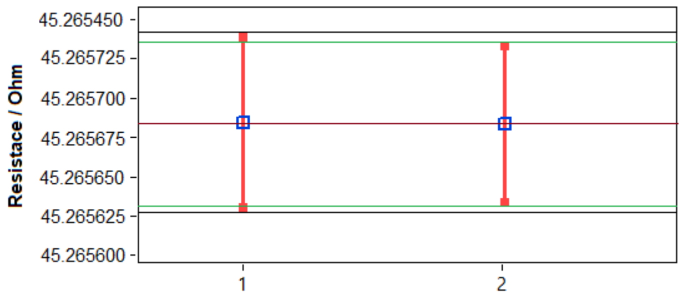


**Figure 12: Uncertainties Comparison between Real Realization (2) and Mathematically Expectation (1)**

**Table 2: Validation of Equations 14 and 17**

| T / K | Experimentally / mK | Statistically / mK | Difference mK |
|---|---|---|---|
| 0.1 | 3.9 | 4.1 | 0.2 |
| 200 | 4.5 | 4.7 | 0.2 |
| 400 | 5.2 | 5.5 | 0.3 |

## 5. CONCLUSIONS

Uncertainty sources has been addressed for all kind of contributions that might affect the true value for both SPRT and RTD. The mathematical model has been developed to estimate the uncertainty either in calibrated point in case of RTD and fixed point for SPRT or uncelebrated points for both kinds of thermometers. The propagation of uncertainty focused in antra- extra eign limits of the ranges. The validation of model has been tested and showed good agreements for two cases of study in real measurements and numerically estimated form mathematical propagation of the equations. The SPRT has been tested at uncelebrated point exactly at 200$^{o}$C, the real realization of the run associated with uncertainty better than 4.3 mk and the mathematical expectation of the uncertainty from the propagation model found to be 4.7 mk.

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